European Biochar Certificate - Guidelines for a sustainable production of Biochar

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Guidelines

European Biochar Certificate

for a sustainable production of biochar

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Impressum

These guidelines are effective as of 1 January 2012 and constitute the basis for biochar certification through the independent, governmental accredited inspection agency q.inspecta.

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EBC foundation | certificate

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Guidelines on the production of biochar

European Biochar Certificate (EBC)

Publisher: European Biochar Foundation

1. Objective of the guidelines and certification

For thousands of years, charcoal has been one of civilisation's basic materials. By far the most common use of charcoal is for cooking, for heating and for smouldering when producing metal tools. However, for centuries charcoal and biochar have also been used for conditioning soils, or as litter (bedding) materials, as medicine and also as a feed additive. In the course of the last century most of this traditional knowledge has been lost and is only now being rediscovered.

Thanks to wide-ranging multidisciplinary research and field trials, the understanding of the biological and chemo-physical processes involved in the use of biochar has made great progress. Thus a major increase in the agricultural use of biochar is to be expected for the next years. Usage ranges from soil conditioning, compost additives and carrier for fertilizers, manure treatment and litter (bedding) materials to silage additives, feed-additives, medical applications and others.

Traditional kiln production of charcoal and biochar without afterburning of pyrolysis gases was unsatisfactory with regard to its carbon efficiency and especially its environmental footprint. Accordingly, those kilns are unsuitable for producing larger biochar amounts to be used in future agriculture. Modern pyrolysis plants and also some types of farmer scale kilns like flame curtain pyrolysis systems are now ready to produce biochar from a large variety of different feedstocks in energy efficient way and without harming the environment. As both, biochar properties and the environmental footprint of its production are very much dependent on the technical control of pyrolysis and the type of feedstocks, a secure control system for its production and analysis needs to be introduced.

The intention of the European Biochar Foundation in issuing these guidelines on how to gain biochar certification is to introduce a control mechanism based on the latest research and practices. The European biochar certificate (EBC) aims to enable and guarantee sustainable biochar production. It is introduced to give customers a reliable quality basis, while giving producers the opportunity of proving that their product meets well-defined quality standards. It further aims at providing a firm state-of-the-art knowledge transfer as a sound basis for future legislation. Finally, it is introduced to prevent and hinder misuse or

dangers from the start, as long as no "special interests" are calling for exceptions (e.g. such as cutting down native forests to produce biochar).

Biochar production technology is currently developing very fast, with more than 500 research projects worldwide looking into biochar properties and interactions. Every month new test results and numerous scientific studies appear on the subject. Every year sees new manufacturers of pyrolysis equipment entering the market and the areas in which biochar and biochar products are used are steadily and rapidly growing. The European biochar certificate is closely linked to this research and technical momentum and will accordingly be revised regularly to take into account the latest findings and developments. Thresholds and test methods will be adapted to reflect the latest findings and, if necessary, re-introduced.

The goal of these guidelines is to ensure control of biochar production and quality based on well-researched, legally backed-up, economically viable and practically applicable processes. Users of biochar and biochar-based products will benefit from a transparent and verifiable monitoring and quality assurance. It is our as well as every biochar user's duty to make sure that a good idea will not be carried into misuse. The certificate was designed to serve this goal.

The European Biochar Certificate is a voluntary industry standard in Europe. In Switzerland it is obligatory for the use of biochar in agriculture.

2. Definition of biochar

Biochar is a heterogeneous substance rich in aromatic carbon and minerals. It is produced by pyrolysis of sustainably obtained biomass under controlled conditions with clean technology and is used for any purpose that does not involve its rapid mineralisation to CO_2 and may eventually become a soil amendment.

Biochar is produced by biomass pyrolysis, a process whereby organic substances are broken down at temperatures ranging from 350°C to 1000 °C in a low-oxygen thermal process. Torrefaction, hydrothermal carbonisation and coke production are further carbonisation processes whose end products cannot however be called biochar under the above definition. Biochars are therefore specific pyrolysis chars characterised by their additional environmentally sustainable production, quality and usage features. Gasification is understood as being part of the pyrolysis technology spectrum and can, if optimized for biochar production, be equally certified under the EBC.

In accordance with the certificate to which these guidelines apply, a differentiation is made between two different biochar grades, each with its own threshold values and ecological requirements: "basic" and "premium".

For gaining the European biochar certificate, the following criteria regarding the biomass feedstock, the production method, the properties of the biochar and the way it is applied have to be met.

3. Feedstock

3.1 Only organic wastes listed in the positive list (Appendix 1) may be used in the production of biochar.

3.2 It must be ensured that all non-organic waste such as plastic, rubber, or electronic scrap has been removed.

3.3 Feedstocks must be free of paint, solvents and other organic or non-organic contaminants.

3.4 When using primary agricultural products, it must be guaranteed that these were grown in a sustainable manner.

3.5 Biochar may only be produced from wood from forests or short rotation forestry plantations if their sustainable management, for example through appropriate PEFC or FSC certification, can be proven.

3.6 Feedstocks used for the production of biochar must not be transported over distances greater than 80 km. An exception is made for pyrolysis additives or special biomasses for use in production tests. [Since the current network of pyrolysis facilities is not yet extensive an exemption to this transport distance requirement can be granted as long as such exemption is only a temporary measure.]

3.7 Complete records of feedstocks must be kept.

4. General requirements for biochar production records

Each biochar series must be clearly labelled and be given a unique identification number for reconstructing the circumstances of production and guaranteeing the quality of the biomasses used. For each biochar series, separate production records are to be kept. Each series must be tested to ensure compliance with the required threshold values.

A uniform biochar series is deemed to exist when the following criteria are met:

1. The pyrolysis temperature in °C do not fluctuate more than 20%. Interruption of the production is allowed as far as the production parameters keep the same after the resumption of production. For small-scale production with a yearly production below 50 t of biochar continuous recording of production temperatures are not requested.
2. The composition of the pyrolysed biomasses does not fluctuate more than 15% based on the type of feedstock listed in the feedstock positive list.
3. The production period of the series does not exceed one year including any interruption of the production.
4. Complete production records must be kept, providing detailed descriptions and dates of any production problems or halts.

Once any one of these four criteria is not met, the biochar subsequently produced belongs to a new series for which new production records have to be kept.

5. Biochar sampling

The biochar samples have to be taken following the procedure described here. The accredited controlling inspector is entitled to take samples and sent to the accredited laboratory or as retain sample to the EBC.

To obtain a biochar sample as representative as possible (in terms of accuracy and precision) of a total lot (batch), it must be taken in a proper way. For this, the following general guidelines have to be followed:

5.1 A biochar lot (batch) subject to sampling must consist of at least the amount of one day of production.
5.2 Before sampling, the whole lot has to be thoroughly mixed 3 times by turning and piling it upside-down by means of physical replacement with a front loader or comparable technical device.
5.3 15 subsamples of 1.5 liter each have than to be arbitrarily gathered from different spots of the homogenized biochar lot (ISO (2006) or Bunge & Bunge (1999)). For small scale production of less than 200 liters per day the subsample size may be reduced to 0.5 liters.
5.4 The 15 subsamples have to be united and milled or crushed if the particle size is above 3 mm.
5.5 The new subsample has than to be homogenized thoroughly by turning and piling it 3 times upside-down.
5.6 A further 15 sub-subsamples of 150 mL each have to be arbitrarily taken from different spots of the gathered subsample lot.
5.7 The 15 sub-subsamples (totaling 2.25 L) have to be united and well mixed.
5.8 The sample of 2.25 L has to be sent to an accredited laboratory for the EBC analyses.

As illustrated in Bucheli et al. (2014), such a sampling procedure my still not be sufficient to obtain truly representative samples, but assures a degree of accuracy (bias) and reproducibility (variance) affordable to compare analytical results with guide values set in this certificate. In case of biochar production by a continuous process, we advise that biochar producers implement and install incremental cross-stream sampling devices as e.g. presented in Gy (2004), allowing for representative sampling of their product.

6. Biochar properties

Current knowledge and the analytical methods are such that it is at present very difficult and expensive to attain a detailed physical-chemical characterisation of biochar. This means that no complete scientific characterisation of the certified biochar can be required. The focus is therefore on guaranteeing compliance with all environmental threshold values and declaring all product properties of relevance for the agricultural use of biochar.

6.1 The biochar's carbon content must be higher than 50% of the dry mass (DM). Pyrolysed organic matter with a carbon content lower than 50% are classified as Pyrogenic Carbonaceous Material (PCM).

The organic carbon content of pyrolysed chars fluctuates between 5% and 95% of the dry mass, dependent on the feedstock and process temperature used. For instance the carbon content of pyrolysed poultry manure is around 25%, while that of beech wood is around 85% and that of bone is less than 10%.
When using mineral-rich feedstocks such as sewage sludge or animal manure, the pyrolysed products tend to have high ash content. Pyrolysed chars with carbon contents below 50% are therefore not classified as biochar but as Pyrogenic Carbonaceous Material (PCM).
When PCM meet all other threshold criteria of this biochar certificate, they may be marketed as EBC certified Pyrogenic Carbonaceous Material (PCM). PCM have a high nutrient content, therefore representing a valuable fertiliser additive. This does, however, mean that they belong to a different product category.
Permitted test methods: DIN 51732
(Specify for each batch)

6.2 The molar H/Corg ratio must be less than 0.7

The molar H/Corg ratio is an indicator of the degree of carbonisation and therefore of the biochar's stability. The ratio is one of the most important characterising features of biochar. Values fluctuate dependent on the biomass and process used. Values exceeding 0.7 are an indication of non pyrolytic chars or pyrolysis deficiencies (Schimmelpfennig and Glaser, 2012).
Permitted test methods: DIN 51732
(Specify for each batch)

6.3 The molar O/Corg ratio must be less than 0.4

In addition to the H/Corg ratio, the O/Corg ratio is also relevant for characterising biochar and differentiating it from other carbonisation products (Schimmelpfennig and Glaser, 2012). Compared to the H/Corg ratio, direct measuring of the O content is relatively expensive and not standardized. Therefore the calculation of the O content from C, H, N, S and ash content is accepted.
Permitted test methods: DIN 51733, ISO 17247
(Specify for each batch)

6.4 The quantity of Volatile Organic Compounds (VOC) must be available and listed.
During the pyrolysis process aromatic carbon, carbonates and a multitude of divers volatile organic compounds are formed. The later constitute a large part of the pyrolysis gas that partly condensates on biochar surfaces and pores. These condensated pyrolysis gas compounds are substantial constituents of biochar materials (Spokas et al., 2011; Yang et al., 2013), are essential for certain biochar functions and thus necessary for the characterisation of biochar and PCM. The VOC-content is further an important indicator for the evaluation of the pyrolysis process.
Permitted test methods: Thermal-Gravimetric-Analysis (TGA)
Principle: The TGA determines the loss of weight of the volatile matter according to the temperature without oxygen.
(Specify for each batch for producers of more than 50 t biochar or PCM per year)

6.5 The biochar nutrient contents with regard to nitrogen, phosphorus, potassium, magnesium and calcium must be available and listed on the delivery slip.
The nutrient contents of different biochars are subject to major fluctuations. For a carbon content exceeding 50%, they can range from 1% to 45%. Please note that these nutrients may only partly be available to plants. They may take decades to enter the biological life cycle. The nutrient availability of the phosphorus found in biochar is for instance only 15% in the first year, that of nitrogen a mere 1%, while that of potassium can reach 50% (Camps-Arbestain et al., 2015).
Permitted test methods: DIN EN ISO 17294 – 2 (E29)
(Specify for each batch)

6.6 The following thresholds for heavy metals must be kept
The following maximum values for heavy metals correspond - for the basic quality grade - to Germany's Federal Soil Protection Act (Bundes-Bodenschutzverordnung or BBodSchV), and - for the premium quality grade - to Switzerland's Chemical Risk Reduction Act (Schweizerische Chemikalien-Risikoreduktions-Verordnung or ChemRRV), Appendix 2.6 on recycling fertilisers. The respective thresholds refer to the biochar's total dry mass (DM):

basic: Pb < 150 g/t DM; Cd < 1,5 g/t DM; Cu < 100 g/t DM; Ni < 50 g/t DM; Hg < 1 g/t DM; Zn < 400 g/ t DM; Cr < 90 g/t DM; As < 13 g t^{-1} TM
premium: Pb < 120 g/t DM; Cd < 1 g/t DM; Cu < 100 g/t DM; Ni < 30 g/t DM; Hg < 1 g/t DM; Zn < 400 g/t DM; Cr < 80 g/t DM; As < 13 g t^{-1} TM

Except some heavy metals that are volatile or semi volatile at the temperature of pyrolysis, the amount of heavy metals contained in the originally feedstock will remain in the final product. As in composting most heavy metals will naturally be more concentrated than in the starting material. However biochar is able to very effectively bind a number of heavy metals, thereby immobilising them for a long period of time. How long has not, however, as yet been determined. As the amounts of biochar used in agriculture are relatively low compared to those of compost and manure, toxic accumulation of heavy metals could practically be ruled out, even when thresholds are higher. Nevertheless this is no sufficient

reason to disregard the heavy metal thresholds stipulated in Germany's Soil Protection Act or Switzerland's Chemical Risk Reduction Act, or any other European legislation.

Abrasion in connection with the use of chromium-nickel steels in the construction of pyrolysis reactors may lead, especially in the first weeks of production, to an increased nickel contamination of biochar. A unique exemption can be granted for biochars with a nickel contamination below 100 g/t DM. Such biochars shall only to be used for composting purposes since the valid thresholds are complied with in the finished compost.
Permitted test methods
Heavy metals: DIN EN ISO17294-2 (E29)
Mercury: DIN EN1483 (E12)
(Specify for each batch)

6.7 The delivery slip must specify the biochar's pH value, bulk density, water and ash content and its specific surface area.

The biochar's pH value is an important criterion with regard to its specific use in substrates, soil amendments, or in binding nutrients in animal husbandry. When a biochar has a pH value exceeding 10, the delivery slip must feature appropriate handling information (regarding health and safety dangers).
Details on bulk density and water content are necessary for the production of homogeneous substrate mixtures or filter ingredients requiring constant carbon contents. The specific surface area is a measure of a biochar's quality and characteristics, and a control value for the pyrolysis method used. It should preferably be higher than 150 m^2/g DM. In some cases lower specific surfaces area than 150 m2/g might, however, be desirable.
The water holding capacity of a given biochar is a valuable indication on it’s effectiveness in increasing a soil's water holding capacity or for humidity buffering in building materials. However, its analysis is not mandatory.
Permitted test methods:
pH: analogous to DIN 10 390
Water content: DIN 51718; TGA 701 D4C
Specific surface area: BET measurement ISO 9277
(Specify for each batch)

6.8 The biochar's PAH content (sum of the EPA's 16 priority pollutants) must be under 12 mg/kg DM for basic grade and under 4 mg/kg DM for premium grade biochar.

As in any combustion, pyrolysis also causes polycyclic aromatic hydrocarbons (PAHs) to be released (Fagernäs et al., 2012). Their amount is dependent in particular on production conditions (Bucheli et al., 2015). Modern pyrolysis methods allow a significant reduction of the PAH pollution. High PAH levels are an indication of unsatisfactory or unsuitable production conditions.
On the other hand, biochar is able to very effectively bind PAHs, with activated biochar being used as an air filter for removing PAHs from exhaust gases and for immobilising PAHs in contaminated soils (Li et al., 2014). The risk of PAH contamination, when using biochar in

agriculture, is hence considered to be low, even if higher thresholds would be taken into account.
Though some PAHs bound in biochar are available to plants, this takes place at an even lower level than with compost, digestate or manure due to biochars' adsorptive capacity (Gomez-Eyles et al., 2013). Moreover, whereas up to 40 tonnes of compost or manure may be applied per hectare over a 3-year period, current reference values for biochar estimate a maximum of 40 to 50 tonnes per hectare over a 100-year period. Total amount of PAH over a 100 years period through such a maximum amount of biochar would thus be at least 30 times less compared to the legal maximum of compost over the same period.
Nevertheless current approval practice indicates that the PAH threshold defined in the Swiss Chemical Risk Reduction Act (ChemRRV) will also apply to biochar and that an exemption on the grounds of biochar's sorption properties is hardly feasible. Therefore, the threshold for premium grade biochar corresponds to the PAH threshold defined in the Swiss Chemical Risk Reduction Act (ChemRRV), also used as a guideline in the Compost Act (Kompostverordnung). No PAH thresholds are specified yet in the European soil protection regulations for soil conditioners and organic fertilisers. The threshold for basic grade biochar is therefore based on a value which, taking the latest research into account, only implies a minimum risk for soils and users.

Please note that, due to biochar's high adsorption properties, most standard methods for testing PAHs are unsuitable for biochar. According to researches carried out by Agroscope ART (Hilber et al., 2012), a longer-term Toluol extraction is needed before any suitably representative test value can be determined. DIN EN 15527: 2008-09 (with toluol extraction) proved to be close to the method of Hilber et al. (2012) and is admitted.
Permitted test methods: DIN EN 15527: 2008-09 (with toluol extraction); DIN ISO 13877: 1995-06 – Principle B with GC-MS; recommended: (Hilber et al., 2012); DIN CEN/TS 16181 (soxhlet-extraction with toluol, analysed by GC-MS or HPLC)
(Specify for each batch)

6.9 PCB content must be below 0.2 mg/kg DM; levels of dioxins and furans must be below 20 ng/kg (I-TEQ OMS).

Modern pyrolysis facilities produce only very low levels of PCB, dioxins and furans, meaning that one control per production unit can be considered sufficient. Dioxin content is mostly dependent on the chlorine content of the feedstock. All authorized feedstock of the feedstock positive list have low chlorine content and will produce during pyrolysis only dioxin contents that are lower than the threshold by several orders of magnitude. If the controlling organism or the EBC considers the risk of chlorine contamination of a given feedstock as relevant, they can require supplemental dioxin analyses. Thresholds are based on the soil protection regulations applicable in Germany and Switzerland (BBodschV, VBBo, ChemRRV).
Permitted test methods: AIR DF 100, HRMS
(Specify for each production unit for producers of more than 50 t biochar or PCM per year)

7. Pyrolysis

7.1 Biomass pyrolysis must take place in an energy-autonomous process.
The external energy used for operating the reactor (electricity for power drive systems, ventilation and automatic control systems, fuel for preheating, etc.) must not exceed 8% (basic grade) or 4% (premium grade) of the calorific value of the biomass pyrolysed in the same period. With the exception of preheating, no fossil fuels are permitted for reactor heating. Industrial waste heat (e.g. from cement or biogas production) or renewable energy sources like solar heat may be used for reactor heating in order to use the syngases for energy production or for motor fuel.

7.2 The pyrolysis gases produced during pyrolysis must be trapped or burned. They are not allowed to escape into the atmosphere.
Most of the global charcoal and biochar production is still done using obsolete technology (Brown et al., 2015) where most of the original feedstock carbon is released as toxic emissions to the atmosphere. Even though the quality of biochar produced in such kilns may meet EBC requirements, the environmental impact of such production techniques is highly negative.
If pyrolysis gases are trapped and used as fuel and bio-oil or are cleanly burned the environmental impact is neutral or even improved compared to biomass burning or natural decomposing. Under the EBC biochar production technology that releases unburned pyrolysis gases are not permitted.

7.3 Syngas combustion must comply with national emission thresholds for such furnaces.
With emission thresholds and regulations differing from one European country to the next, any further definition of emission thresholds for pyrolysis facilities would exceed the purpose and proportionality of these guidelines. Therefore manufacturers must provide a guarantee that their facilities comply with national emission regulations.

7.4 The heat produced by the reactor must be recycled.
35 to 60 % of the energy contained in the biomass feedstock can be found in the syngas at the end of pyrolysis. Its combustion can be used for heating the biomass, whereby additional waste heat is produced. At least 70% of this must be used for drying biomass, for heating, for generating electricity or for similar sustainable purposes. Syngas can also be stored and used later for subsequent energy purposes.
Although it is highly recommended, small-scale biochar production units with an annual output of less than 50 tons are exempt of the heat recovery.

8. Sale and application of biochar

8.1 Fire and dust protection regulations are to be complied with throughout the production, transport and end-user chain.

8.2 During transport and bulk transfers attention must be paid to the biochar being sufficiently moist to prevent dust generation or dust explosions. The moisture content of the delivered biochar has to be given to ensure that the customer knows how much dry biochar he/she obtains.

8.3 Production workers must be equipped with suitable protective clothing and breathing apparatus.

8.4 Delivery slips must contain well-visible usage instructions and health and safety warnings.

8.5 When applying biochar on fields or in animal stables and housings, biochar must be kept slightly moist to prevent dust formation. In this respect, producers must provide appropriate information on the delivery slip or on packaging.

9. Quality assurance and certification

Biochar producers' compliance with European Biochar Certificate requirements is coordinated throughout Europe by the independent, governmental accredited quality assurance agency q.inspecta, with inspections of production plants in individual countries carried out by independent national inspection agencies. Inspections take place once a year. Producers confirm that they will keep up-to-date production records.

In order to be reasonably proportional to the risk assessment and to the environmental protection goals, producers with an annual production capacity below 50 t of biochar are exempt from on-site inspection of production. The compliance with production requirements is controlled by the accredited quality assurance agency via self-declaration and a detailed description of the complete production process. The requirements for biochar batch analyses, thresholds, feedstock sustainability and handling of biochar maintain the same as for industrial producers.

Producers may submit applications to q.inspecta to take part in the certification programme once their production starts. They are recommended to contact q.inspecta beforehand, enabling them to integrate the necessary recording into their production process.

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10. Analytical Methods

Basic Package

Sample preparation (DIN 51701-3):
After homogenization, the sample is divided representatively into portions. This subsampling is done by quartering (quarter method) of the homogenized sample. Approximately 100 g of the original sample are used for the determination of the conductivity, the salt content and pH.
A portion of the sample is dried at 40 ° C and is divided into some subsamples after drying and homogenization. Approximately 250 g of the 40 ° C dried and uncrushed sample is used to determine the true density and the BET surface of the material. Approximately 50 g of the 40 ° C dried sample is finely ground in a vibratory mill. After homogenization the fine material is subsampled for further analysis (PAK, TGA, ash, CHN, S, trace and major elements).

Bulk density (analogue VDLUFA-Method A 13.2.1):
The sample (at least 300 ml) is filled into a graduated cylinder and the mass is determined by weighting. The volume of the sample is read after 10 times compression by means of falling. The density in kg / m³ is calculated from the mass and the volume of the sample.

Electrical conductivity (salt content) - Method of the BGK (Federal quality community compost), volume 1, method III. C2 – in analogy to DIN ISO 11265:
Adding 20 g of the sample to 200 ml desalinated water and shaking it for 1 hour, followed by filtration of the solution. The conductivity is measured then in the filtrated water. The correction of temperature is automatically done in the measuring device. The electrical conductivity is given for a solution at 25°C. The salt content is calculated using the factor 52,8 [mg KCl/l]/[10^{-4}/cm] and is given in mg KCl/l. This is based on the conductivity (14,12 * 10^{-4} S/cm) of a 0,01 molar KCl solution.

pH-value DIN ISO 10390 ($CaCl_2$):
Minimum 5 ml of the air-dried sample is placed in a glass vessel. Five times the volume (25 ml) of a 0.01 M $CaCl_2$ solution is added. The suspension is overhead rotated for 1 h. The suspension obtained is directly measured with a pH meter.

Water content according to DIN 51718:
Method A / two-step method (Reference method for coal)

1. raw moisture

The sample (100 to 1000 g) is spread evenly in a drying bowl crucible, weighed with 0,1 g accuracy and dried in an oven at (40 ± 2) ° C until the mass is constant. If necessary, the sample is divided and dried in more than one crucible.

Analysis: raw moisture (FG) in%

$$FG = \frac{m_E - m_R}{m_E} * 100$$

FG = raw moisture in %

m_E = mass of the sample before drying in g

m_R = mass of the sample after drying in g

2. hygroscopic moisture

A subsample of the air-dried and crushed (grain size < 1 mm) sample is weighed immediately after the subsampling into a TGA crucible and is dried in a nitrogen atmosphere at (106 ± 2) ° C to constant mass.

Evaluation: hygroscopic moisture (FH) in %

$$FH = \frac{m_E - m_R}{m_E} * 100$$

FH = hygroscopic moisture in %

m_E = mass of the sample before drying in g

m_R = mass of the sample after drying in g

3.water content

Evaluation: water content (Wt) in %

$$W_t = FG + FH * \frac{100 - FG}{100}$$

W_t = water content in %

FG = raw moisture in %

FH = hygroscopic moisture in %

Ash content (550 °C) analogue DIN 51719:

To determine the ash content two programs of the TGA (30 or 60 min) could be used. The weight determination of the crucible is carried out automatically. Enter the sample number for corresponding crucible position. Add 1,0 g of the sample in the ceramic crucible and spread the substance evenly in the crucible. The weighing is done automatically relative to the crucible position.

The oven runs the following heating program:

- heating with a rate of 5 K / min to 106 ° C under a nitrogen atmosphere to constant mass (Dm <0,05%).
- temperature increase with 5 K / min to 550 ° C under oxygen atmosphere,
- hold this temperature for 30 or 60 min to constant mass (Dm <0,05%).

The ash content is automatically determined and calculated for the used sample.

Thermogravimetry:
The TGA curve is determined, like the ash content, with the LECO TGA. For this purpose, 1,0 g of pre-dried and ground sample is weighed in the TGA crucible. During the temperature rise from 30 ° C to 950 ° C with 10 K / min, the crucible is weighed at frequent intervals in the TGA furnace. The result is shown graphically.

Carbonate CO_2 analogue DIN 51726:
1 g of pre-dried and ground sample is weighed to 0.2 mg and placed in the decomposition flask. The device consists of an absorption tower, which frees the air of carbon dioxide, the decomposition flask with an attachment to add the decomposition acid and three connected washing bottles. The carbon dioxide freed air is sucked through the system. After the system purged and the washing bottles were filled with an absorbing solution of $BaCl_2$ and NaOH solution, 30 ml decomposition acid (hydrochloric acid with $HgCl_2$ as a catalyst and a wetting agent) are added to the decomposition flask. The content of the decomposition flask is boiled for about 10 minutes. The inert gas flow transports the carbon dioxide produced through the acidic solution in the first wash bottle in the other two wash bottles. In the second wash bottle, the carbon dioxide dissolves under consumption of base and is precipitated as barium carbonate. If something precipitates in the third wash bottle, the measurement must be repeated with a lower initial mass. The consumption of base in the second wash bottle is determined by a pH-titration using hydrochloric acid. The carbonate content of the sample is calculated from the base consumption and is calculated as CO_2.

CHN according to DIN 51732:
A TruSpec CHN (Manufacturer: Leco) is used.
The sample (80-100 mg of the pre-dried and crushed sample) is weighed directly (relative precision 0,1%) into a tin capsule. After that the capsule is closed and is put in the machine for measurement. The TruSpec CHN determines the carbon content, the hydrogen content and the nitrogen content in mass percent.

Sulfur according to DIN 51724-3:
The pre-dried and crushed sample is weighed in a ceramic crucible. With the aid of a catalyst layer of V_2O_5 and at high temperatures (> 1300 ° C) the sulfur is oxidized in an oxygen stream. The resulting SO_2 is detected in an IR cell and is calculated with the sample mass as total sulfur content.

Oxygen (calculation) according to DIN 51733:
The oxygen content is a calculated parameter. It is assumed that the sample consists essentially of ash, carbon, hydrogen, nitrogen, sulfur and oxygen. If one subtracts the ash, carbon, hydrogen, nitrogen and sulfur content in percent from 100 %, the result will be the oxygen content in percent.

C_{org}, H/C und O/C (calculation):
Other quantities and ratios can be calculated from the determined data.
C_{org} is derived from the total carbon content minus the inorganic carbon content (CO_2) in the sample.

PAH analogue to DIN EN 15527 (extraktion with Toluol) GC-MS (DIN CEN/TS 16181):
2,5 g of the pre-dried and crushed sample is weighed into a extraction thimble and is extracted with 50 ml of toluene at reflux for two hours. The extract is concentrated to 10 ml. An aliquot of the extract is transferred to an injection vial and the PAH are analyzed by gas chromatography.

Gas chromatograph: Network GC System 7890N and 5975C MSD and inertXL
AS 7693 Fa: Agilent Techn
Capillary column: HP 5MS (30 mx 0.25 mm x 0.25 microns)
Temperature program: 90 ° C (0.5 min), 20 ° C / min to 250 ° C, 5 ° C / min to 275 ° C, 20 ° C / min
to 320 ° C for 5 min
Transfer line: 280 ° C
MSD temperature: 150 ° C
Injection volume: 1 µl
Injector temperature: 250 ° C
Carrier gas: helium (1,5 ml / min)

Trace metals after microwave-assisted digestion according to DIN 22022-2, DIN 22022-7, DIN EN ISO 17294-2 / DIN EN 1483:
(Pb, Cd, Cu, Ni, Hg, Zn, Cr, B, Mn, As)
The pre-dried and crushed sample is weighed into the reaction vessel of the microwave. 6 ml of nitric acid, 2,0 ml of hydrogen peroxide and 0,4 ml of hydrofluoric acid are added. The reaction vessel is sealed and is placed in the microwave.
Program flow of the microwave pressure digestion:
heating (room temperature to 190 ° C) in 15 min
holding time at 190 ° C for 20 minutes
free cooling

additional only for ICP-OES:
Program flow of the fluoride masking (Boric acid, adding 5 ml of saturated solution):
heating (room temperature to 160 ° C) in 8 minutes
holding time at 160 ° C for 7 minutes
free cooling
After complete cooling, the reaction vessels are opened and the digestion solution is transferred to in a 50 mL plastic volumetric flask and filled with deionized water.
The diluted solution is measured by ICP-MS (DIN EN ISO 17294-2).
To determine the levels of mercury a cold vapor AAS (DIN EN 1483) is used.

Main elements after melting digestion DIN 51729, DIN EN ISO 11885 / DIN EN ISO 17294-2: (P, Mg, Ca, K, Na, Fe, Si, S)
The melting process is performed on the ashes of the biochar. 200 mg of the fine ash are weighed into a platinum crucible and thoroughly mixed with 2 g of lithium metaborate. The platinum crucible is placed in a digestion oven. The digestion remains at least 15 minutes at 1050 ° C in the oven. The melt is dissolved in hydrochloric acid and filled to 500 ml.
The samples are measured with ICP-OES (DIN EN ISO 11885) or ICP-MS (DIN EN ISO 17294-2).

Additional parameters

Gross calorific value / net calorific value according to DIN 51900:
For the determination of the calorific values a bomb calorimeter (IKA calorimeter C 5000 duo IKA C 5000) is used. 0,3 to 0,8 g of pre-dried and ground sample is weighed into a combustion bag, capsule or crucible. The sample is mounted in the combustion bomb with an ignition wire and 10-20 ml of eluent in bottom part of the bomb. The bomb is placed into the calorimeter. The oxygen filling, the ignition and the measurement are done automatically. After the combustion the bomb must be checked for signs of incomplete combustion. The gross calorific value is calculated using the calibration and measurement data. With further corrections, the net calorific value is calculated.

Ash content (815 °C) DIN 51719:
The ash content (815 ° C) is determined after the ash content (550 ° C) by rising the temperature from 550 ° C with 5 K / min to 815 ° C and holding until constant weight (mass difference ± 0,05%) is reached.

Volatile matter according to DIN 51720:
1,0 g of the pre-dried and ground sample is weighed into a crucible (with lid). The sample must form a uniformly thick layer on the bottom of the crucible. The crucible is placed in the oven preheated at 900 ± 5 ° C. After 7 minutes (± 5 sec), the crucible is removed from the oven and reweighed after cooling to room temperature. The volatile matter content is calculated from the mass loss of the sample.

Water holdung capacity (WHC) according to DIN ISO 14238-2011
Water-holding capacity. This can be measured using the method according to German Standard E DIN ISO 14238-2011; appendix A (draft).
The test consists of soaking the 2mm fraction of the material in water for a period of 24 hours. After this, the material has to be placed on a dry sand bed for 2 hours for removing free water. The saturated material has to be weighed and then dried at 40°C in a compartment dryer. After drying the material has to be weighed again for estimate the water holding capacity.

11. References

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